Technical Report
1175

Measurement of the Absolute Raman Cross Sections of Diethyl Phthalate, Dimethyl Phthalate, Ethyl Cinnamate, Propylene Carbonate, Tripropyl Phosphate, 1,3-Cyclohexanedione, 3'-Aminoacetophenone, 3'-Hydroxyacetophenone, Diethyl Acetamidomalonate, Isovanillin, Lactide, Meldrum's Acid, p-Tolyl Sulfoxide, and Vanillin

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13 September 2013

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JPEO-CBD security has reviewed this report, and it is releasable to the National Technical Information Service, where it will be available to the general public, including foreign nationals.

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ABSTRACT

We have measured the Raman spectra of several chemicals for 785-nm excitation using a Raman spectrometer with focal length of 500 mm, f-number of 6.5, and resolution of 2.0 ± 0.1 cm^{-1}. Values of $1.92 \pm 0.12 \times 10^{-29}$, $2.50 \pm 0.15 \times 10^{-29}$, $3.10 \pm 0.19 \times 10^{-28}$, $1.17 \pm 0.07 \times 10^{-29}$, and $1.53 \pm 0.09 \times 10^{-29}$ cm^2 have been determined for the Raman cross section σ_R of neat Diethyl phthalate (DEP), dimethyl phthalate (DMP), ethyl cinnamate (EC), propylene carbonate (PC), and tripropylene phosphate (TPP) for the Raman modes at 1041, 1040, 1638, 849, and 726 cm^{-1}, respectively. The values of the linewidth (FWHM) Γ for these modes have been determined to be 6.6 ± 0.1, 7.0 ± 0.1, 11.3 ± 0.2, 15.8 ± 0.2, and 29.0 ± 0.3 cm^{-1}, respectively. Values of $1.47 \pm 0.12 \times 10^{-28}$, $1.67 \pm 0.13 \times 10^{-29}$, $5.15 \pm 0.40 \times 10^{-29}$, $3.25 \pm 0.26 \times 10^{-29}$, $1.57 \pm 0.13 \times 10^{-28}$, $2.07 \pm 0.17 \times 10^{-29}$, $1.00 \pm 0.08 \times 10^{-29}$, $3.73 \pm 0.30 \times 10^{-29}$, and $1.42 \pm 0.11 \times 10^{-29}$ cm^2 have been determined for σ_R for the powders of 1,3-cyclohexanedione (CHD), 3'-aminoacetophenone (AAP), 3'-hydroxyacetophenone (HOAP), diethyl acetamidomalonate (DEAM), isovanillin (ISOV), lactide (DODO), Meldrum's acid (MA), p-tolyl sulfoxide (PTSO), and vanillin (VAN) for the Raman modes at 1174, 714, 1663, 2943, 1671, 657, 641, 796, and 814 cm^{-1}, respectively. The values of Γ for these modes have been determined to be 8.4 ± 0.2, 4.4 ± 0.1, 4.5 ± 0.1, 10.6 ± 0.2, 4.5 ± 0.1, 2.7 ± 0.1, 6.8 ± 0.2, 5.7 ± 0.2, and 5.2 ± 0.1 cm^{-1}, respectively.

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The authors wish to thank Dr. T.Y. Fan for his comments regarding this report.

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1. INTRODUCTION

The values of the absolute Raman cross sections σ_R of solids, liquids, and gases are important for the science and technology of these materials. Values of the absolute σ_R of relatively few materials have been reported. These include benzene [1,2], benzenethiol [3], bis-2-chlorethyl sulfide (HD) [4], carbon monoxide [5], carbon dioxide [5], diamond [6–8], diisopropyl methylphosphonate (DIMP) [4], dimethyl methylphosphonate (DMMP) [4], ethyl dimethylphosphoramidocyanidate (GA) [4], ethyl S-2-diisopropylamino ethylmethylphosphonothioate (VX) [4], gallium phosphide [9,10], hydrogen sulfide [5], isopropyl methylphosphonofluoridate (GB) [4], nitrogen [5], oxygen [5], and silicon [11,12].

Here, we report the measurement of the absolute Raman cross sections of fourteen materials, which substantially expands the number of materials for which such measurements have been made. These are diethyl phthalate (DEP), dimethyl phthalate (DMP), ethyl cinnamate (EC), propylene carbonate (PC), 1,3-cyclohexanedione (CHD), 3'-aminoacetophenone (AAP), 3'-hydroxyacetophenone (HOAP), diethyl acetamidomalonate (DEAM), isovanillin (ISOV), lactide (DODO),[1] Meldrum's acid (MA),[1] p-tolyl sulfoxide (PTSO), tripropyl phosphate (TPP), and vanillin (VAN).

[1] Lactide and Meldrum's acid are synonyms for 3,6-dimethyl-1,4-dioxane-2,5-dione and 2.2-dimethyl-1,3-dioxane-4,6-dione, respectively.

2. EXPERIMENTAL

A schematic of the optical setup for the measurement of Raman cross sections is shown in Figure 1.

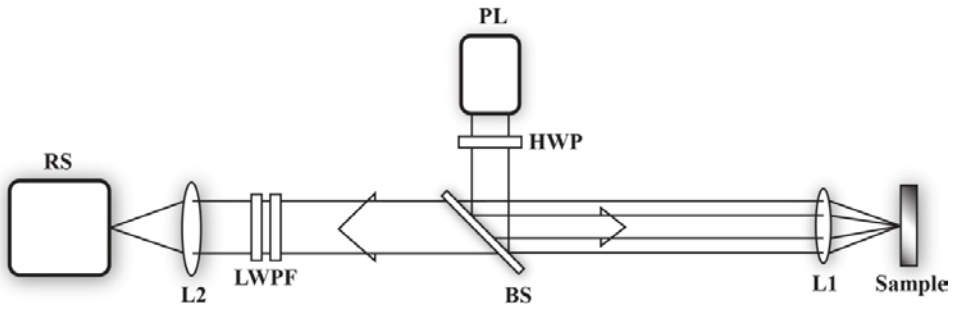

Figure 1. Schematic of the optical setup used for the measurement of the Raman cross sections.

The acronym PL is for pump laser. HWP is a half-wave plate for setting the polarization of the pump laser. BS is a beamsplitter for reflecting the pump laser beam towards the sample and for transmitting the backscattered Raman light. L1 is a lens for focusing the pump laser on the sample and for the collimation of the Raman scattered light. LWPFs are long-wave-pass filters for blocking the pump light. L2 is a lens for focusing the Raman light on the entrance slit of the Raman spectrometer (RS).

The pump laser is a 784.9-nm, 1400-mW cw single-mode (both transverse and longitudinal), linearly polarized Sacher Lasertechnik diode laser model SYS-420-0785-1400 (Sacher Lasertechnik, Marburg, Germany/Buena Park, California). The HWP is Edmund Optics part number 46-561 (Edmund Optics, Barrington, New Jersey). The BS is Semrock part number LPD01-785RS-25×36×1.1 with 803–1214 nm passband (Semrock, Rochester, New York). The lens L1 is a 12-mm-diameter, 20-mm-effective-focal-length (EFL), near-infrared (NIR) achromat Edmund Optics part number 45-792. The long-wave-pass filters are 25-mm-diameter Semrock part number LP02-785RE-25 with 790–1770 nm passband. The lens L2 is a 25-mm-diameter, 40-mm-EFL, NIR achromat Edmund Optics part number 45-801. The Raman spectrometer is a 500-mm-focal-length, f# 6.5, Princeton Instruments model SP-2556, which is equipped with the Princeton Instruments camera PIXIS 100BR (Princeton Instruments, Trenton, New Jersey). The grating in the spectrometer has 1200 grooves/mm. The blaze wavelength of the grating is 750 nm.

All the chemicals used in this work were purchased from Sigma-Aldrich. The values of the molecular mass M and density ρ of these materials are given in Table 1. The values of ρ for the liquids

are obtained from their Material Safety Data Sheets (MSDS). The values of ρ of the powders were deduced from the measured mass and volume contained in the near infrared (NIR) 1.0-mm path length optical cells, Spectrocell part no. R-4001-T (Spectrocell Inc., Oreland, Pennsylvania) with fused silica windows.

Table 1

Values of the Molecular Mass M and Density ρ Along with CAS Number and Chemical Formula

Material	CAS No.	Phase	Chemical Formula	M (g/mol)	ρ (g/cm^3)
Diethyl phthalate (DEP)	84-66-2	Liquid	$C_{12}H_{14}O_4$	222.24	1.12
Dimethyl phthalate (DMP)	131-11-3	Liquid	$C_{10}H_{10}O_4$	194.18	1.19
Ethyl cinnamate (EC)	103-36-6	Liquid	$C_{11}H_{12}O_2$	176.21	1.05
Propylene carbonate (PC)	108-32-7	Liquid	$C_4H_6O_3$	102.09	1.19
Tripropyl phosphate (TPP)	513-08-6	Liquid	$C_9H_{21}O_4P$	224.23	1.01
1,3-Cyclohexanedione (CHD)	504-02-9	Powder	$C_6H_8O_2$	112.13	0.29
3'-Aminoacetophenone (AAP)	99-03-6	Powder	C_8H_9NO	135.16	0.54
3'-Hydroxyacetophenone (HOAP)	121-71-1	Powder	$C_8H_8O_2$	136.15	0.50
Diethyl acetamidomalonate (DEAM)	1068-90-2	Powder	$C_9H_{15}NO_5$	217.22	0.74
Isovanillin (ISOV)	621-59-0	Powder	$C_8H_8O_3$	152.15	0.78
Lactide (DODO)	95-96-5	Powder	$C_6H_8O_4$	144.13	0.81
Meldrum's acid (MA)	2033-24-1	Powder	$C_6H_8O_4$	144.13	0.58
p-Tolyl sulfoxide (PTSO)	1774-35-2	Powder	$C_{14}H_{14}OS$	230.33	0.39
Vanillin (VAN)	121-33-5	Powder	$C_8H_8O_3$	152.15	0.51

The purity of the chemicals used in this work is as follows: DMP 99.5%, DEP 99.9%, EC 99.7%, PC 99.97%, CHD >99.9%, AAP 99.3%, HOAP 99.9%, DEAM 99.9%, ISOV 99.96%, DODO 99.60%, MA >99.9%, PTSO 98.2%, TPP 99.7%, and VAN 99.9%.

3. THEORETICAL

The Raman cross section σ_R (cm^2) of a Raman mode ν_R is given by

$$\sigma_R = \left(\frac{4\pi}{K}\right)\left(\frac{M}{\rho N_A}\right)\left(\frac{N_R}{N_P}\right), \tag{1}$$

where K is a constant that takes into account the transmittance of the L1 lens (93%) and the fused silica front window of the optical cell (96.5%) for the pump light and the Raman scattered light, and the transmittance of the beamsplitter (98%), four LWPFs (92%), and the L2 lens (93%) for the Raman scattered light. The value of K has been determined to be 0.68 ± 0.02. M (g/mol) is molecular mass, ρ (g/cm^3) is the density, N_A is the Avogadro constant, N_R is the number of Raman photons ($s^{-1}\cdot cm^{-1}\cdot sr^{-1}\cdot mW^{-1}$) integrated over the Raman mode, and N_P is the number of pump photons ($s^{-1}\cdot mW^{-1}$) equal to 3.96×10^{15}. Using the values of K, N_A, and N_P, Eq. (1) may be written as

$$\sigma_R = 7.75 \pm 0.11 \times 10^{-39}\left(\frac{MN_R}{\rho}\right). \tag{2}$$

We neglect the effect of the elastic scattering of the pump light on the measurement of the Raman cross section.

4. RESULTS AND DISCUSSION

Figures 2 through 6 show the Raman spectra (Raman signal/data point versus Raman shift) of neat DEP, DMP, EC, PC, and TPP. Data-point spacing is 0.42 to 0.20 cm^{-1} for the 200 to 3200 cm^{-1} Raman shift.

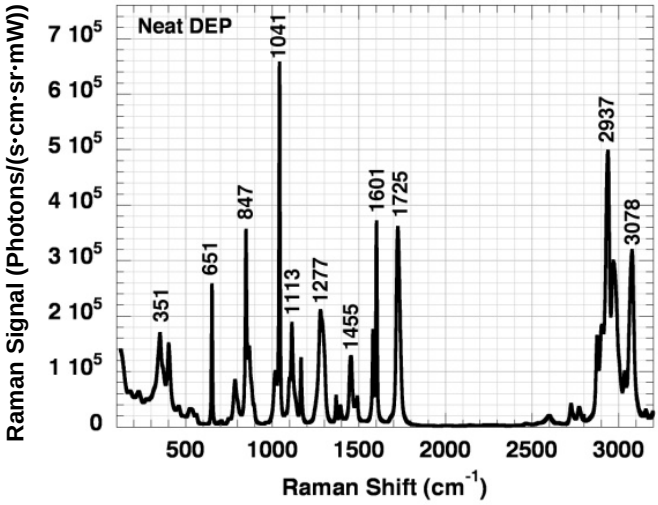

Figure 2. Raman spectrum of neat diethyl phthalate (DEP) obtained using 100 mW of 785-nm pump power.

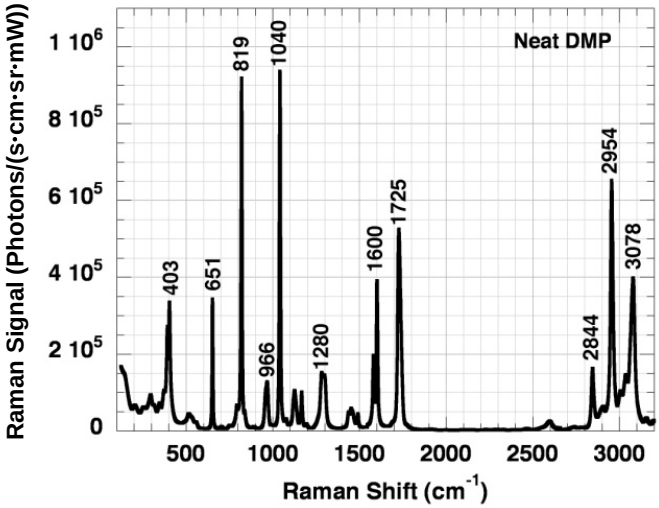

Figure 3. Raman spectrum of neat dimethyl phthalate (DMP) obtained using 100 mW of 785-nm pump power.

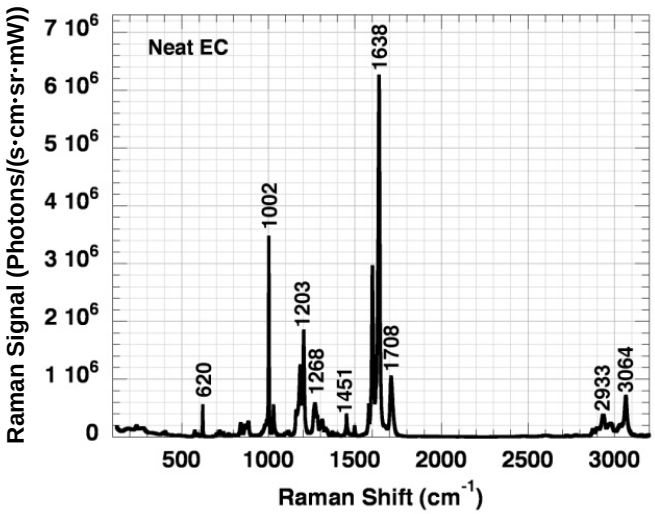

Figure 4. Raman spectrum of neat ethyl cinnamate (EC) obtained using 100 mW of 785-nm pump power.

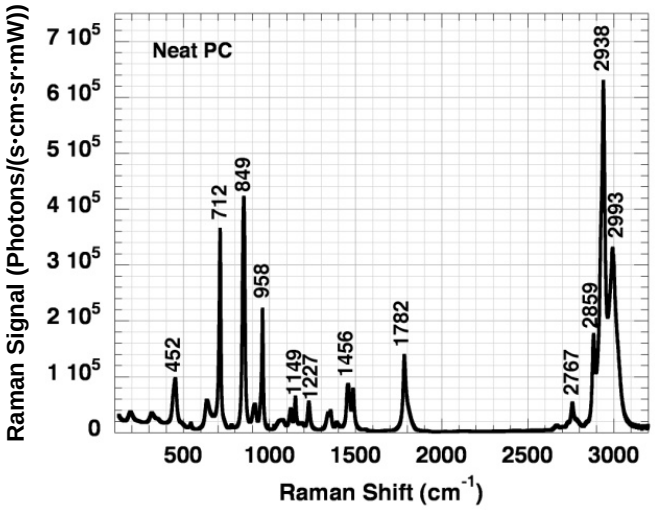

Figure 5. Raman spectrum of neat propylene carbonate (PC) obtained using 100 mW of 785-nm pump power.

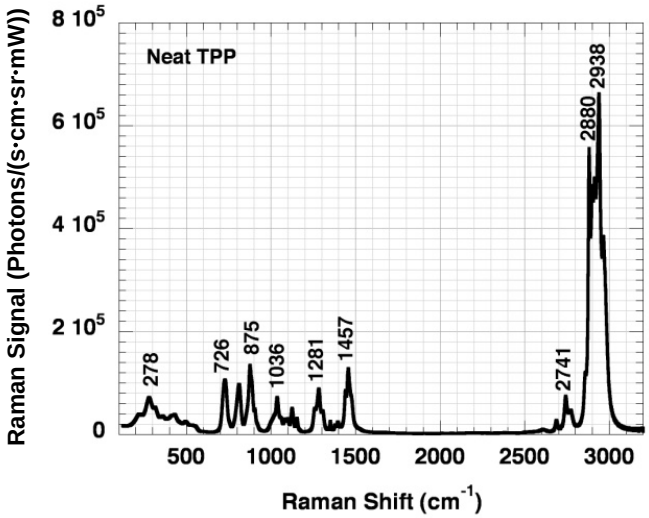

Figure 6. Raman spectrum of neat tripropyl phosphate (TPP) obtained using 100 mW of 785-nm pump power.

All the Raman spectra were measured using 100 mW of the pump power incident upon the lens L1. Each spectrum is the average of three scans for the 125 to 3200 cm^{-1} range, which consists of nine segments. Each segment is the average of 20 frames with each frame obtained with an integration time of 1.0 s. Thus, it takes 9 minutes for the acquisition of each spectrum. Using the 785-nm pump laser, the spectral resolution of the Raman spectrometer (FWHM) was determined to be 2.0 ± 0.1 cm^{-1}.

The values of N_R for the 1041-, 1040-, 1638-, 849-, and 726-cm^{-1} modes of DEP, DMP, EC, PC, and TPP are $1.25 \pm 0.06 \times 10^7$, $1.98 \pm 0.10 \times 10^7$, $2.38 \pm 0.12 \times 10^8$, $1.76 \pm 0.09 \times 10^7$, and $8.87 \pm 0.44 \times 10^6$ photons ($s^{-1} \cdot cm^{-1} \cdot sr^{-1} \cdot mW^{-1}$), respectively; the values of N_R were obtained after subtracting the background. The values of σ_R have been determined by substituting the values of M, N_R, and ρ in Eq. (2). The accuracy of σ_R for neat DEP, DMP, EC, PC, and TPP is estimated to be ±6%, which is due to the ±5%, ±3%, and ±1.0% uncertainty in the values of N_R, K, and ρ, respectively. The values of the linewidth Γ (full width at half maximum) and σ_R for neat DEP, DMP, EC, PC, and TPP are given in Table 2.

Figures 7 through 15 show the Raman spectra of the CHD, AAP, HOAP, DEAM, ISOV, DODO, MA, PTSO, TPP, and VAN powders.

Table 2

Values of the Linewidth Γ (FWHM) and Raman Cross Section σ_R for the 1041-, 1040-, 1638-, 849-, and 726-cm^{-1} Modes of Neat DEP, DMP, EC, PC, and TPP, Respectively

Material	Mode (cm^{-1})	Γ (cm^{-1})	σ_R (cm^2)
DEP	1041	6.6 ± 0.1	$1.92 \pm 0.12 \times 10^{-29}$
DMP	1040	7.0 ± 0.1	$2.50 \pm 0.15 \times 10^{-29}$
EC	1638	11.3 ± 0.2	$3.10 \pm 0.19 \times 10^{-28}$
PC	849	15.8 ± 0.2	$1.17 \pm 0.07 \times 10^{-29}$
TPP	726	29.0 ± 0.3	$1.53 \pm 0.09 \times 10^{-29}$

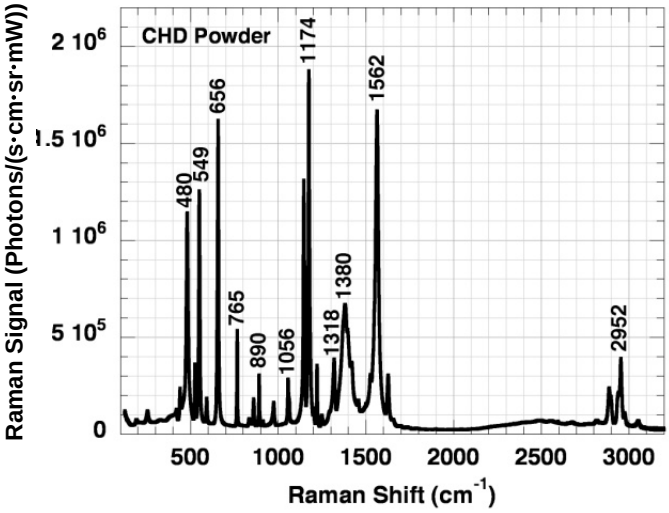

Figure 7. Raman spectrum of 1,3-cyclohexanedione (CHD) powder obtained using 100 mW of 785-nm pump power.

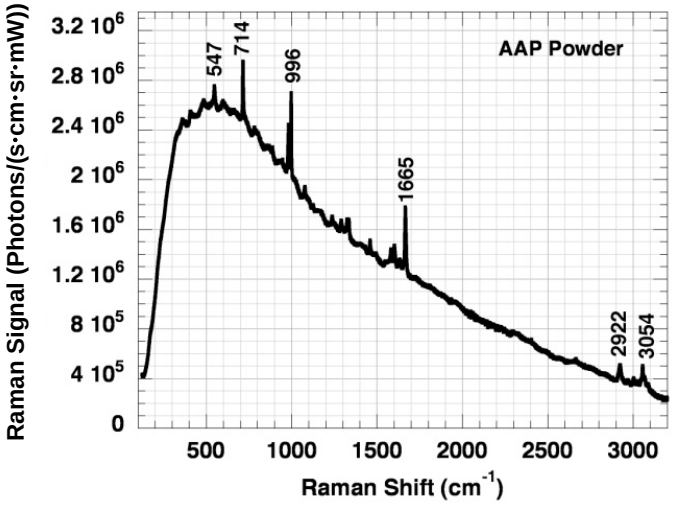

Figure 8. Raman spectrum of 3'-aminoacetophenone (AAP) powder obtained using 100 mW of 785-nm pump power.

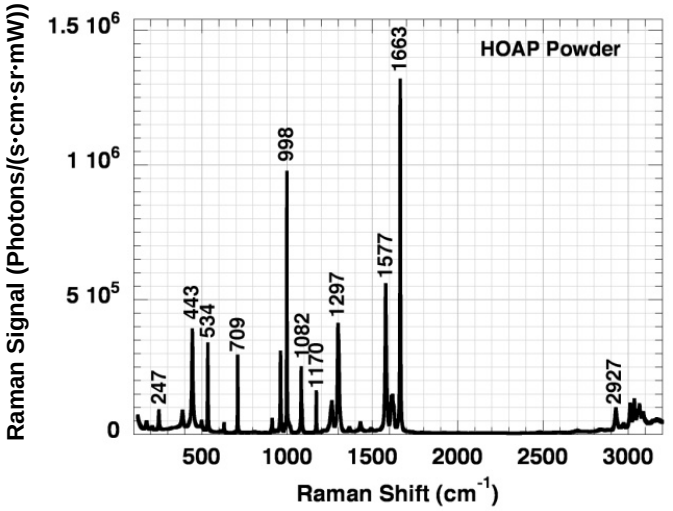

Figure 9. Raman spectrum of 3'-hydroxyacetophenone (HOAP) powder obtained using 100 mW of 785-nm pump power.

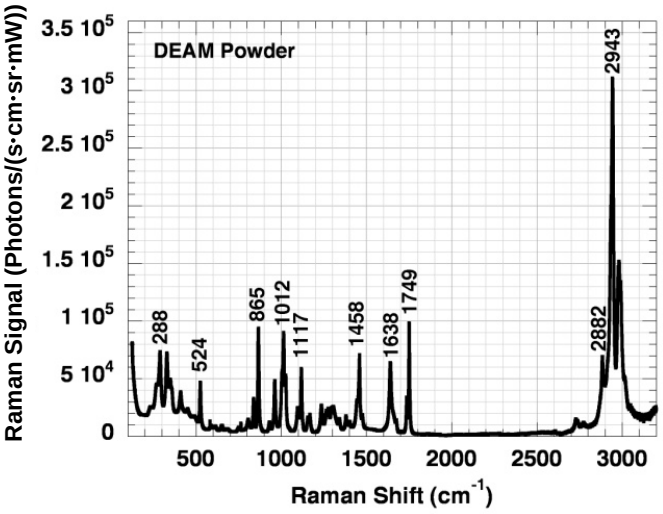

Figure 10. Raman spectrum of diethyl acetamidomalonate (DEAM) powder obtained using 100 mW of 785-nm pump power.

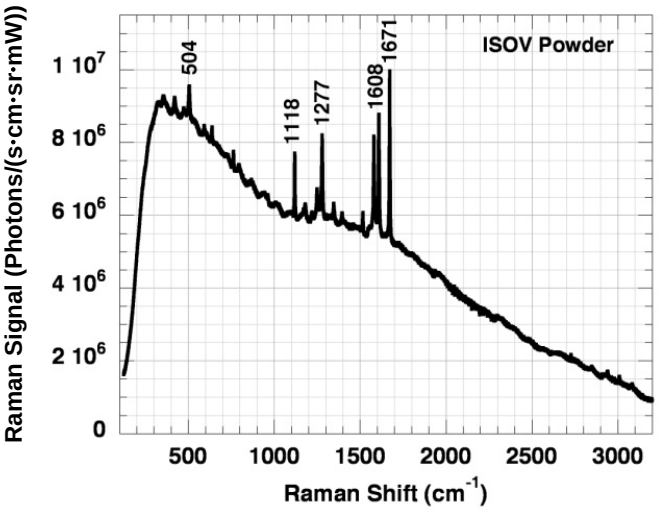

Figure 11. Raman spectrum of isovanillin (ISOV) powder obtained using 100 mW of 785-nm pump power.

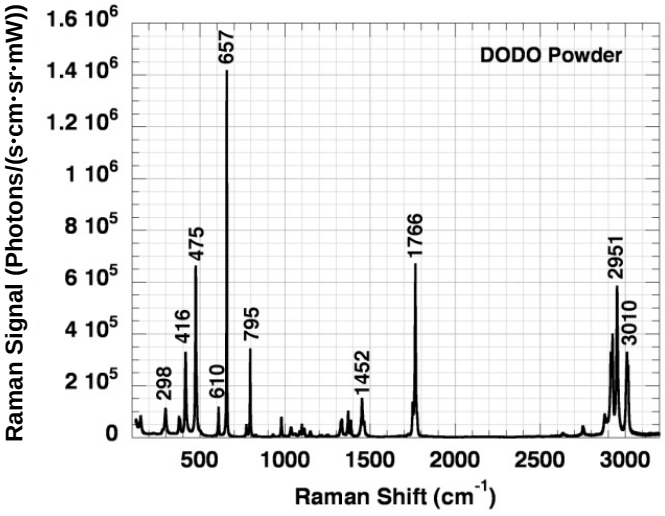

Figure 12. Raman spectrum of lactide (DODO) powder obtained using 100 mW of 785-nm pump power.

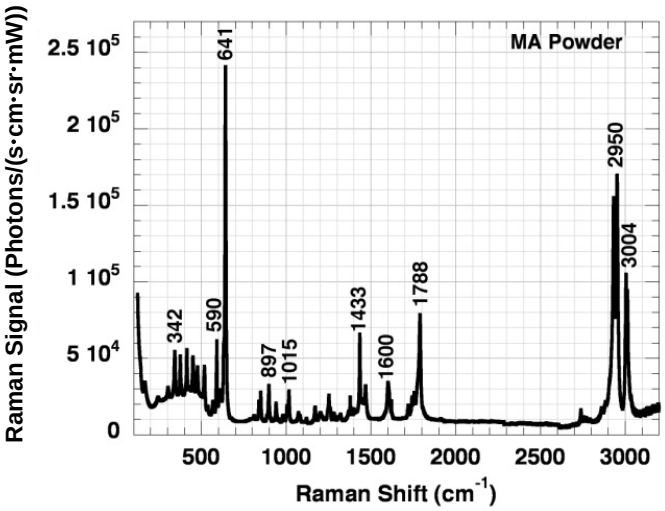

Figure 13. Raman spectrum of Meldrum's acid (MA) powder obtained using 100 mW of 785-nm pump power.

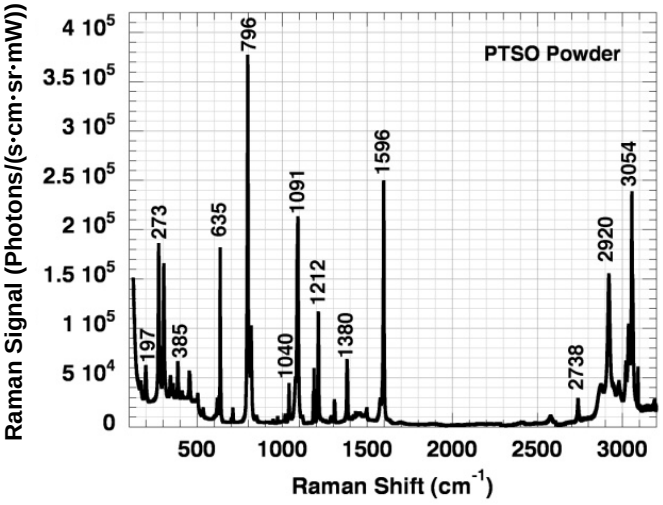

Figure 14. Raman spectrum of p-tolyl sulfoxide (PTSO) powder obtained using 100 mW of 785-nm pump power.

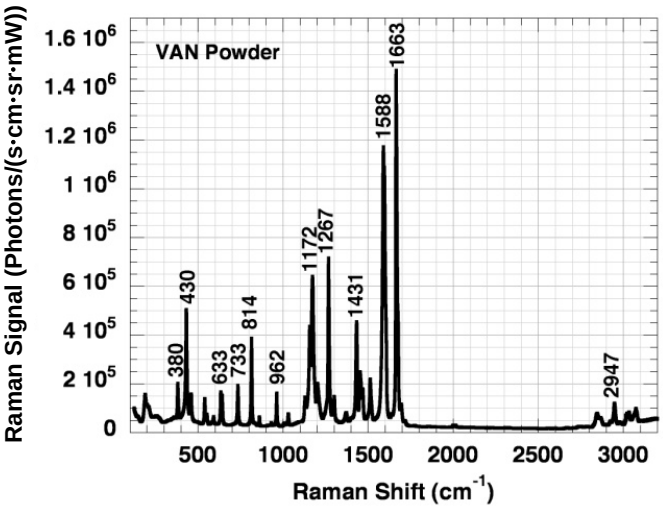

Figure 15. Raman spectrum of vanillin (VAN) powder obtained using 100 mW of 785-nm pump power.

The large background in the Raman spectra of AAP and ISOV in Figures 8 and 11, respectively, is most likely due to fluorescence. The values of N_R were obtained after subtracting the background. The accuracy of σ_R of the powders is estimated to be ±8%, which is due to the ±5%, ±3%, and ±5.0%

uncertainty in the values of N_R, K, and ρ, respectively. The effect of the elastic scattering of the pump beam, neglected in this work, may substantially decrease the estimated accuracy of σ_R. Also, the values of σ_R obtained in this work represent the lower bound because of the neglect of the elastic scattering of the pump beam. The values of Γ, N_R, and σ_R for CHD, AAP, HOAP, DEAM, ISOV, DODO, MA, PTSO, and VAN are given in Table 3.

Table 3

Values of the Linewidth Γ (FWHM), N_R, and Raman Cross Section σ_R for the 1174-, 714-, 1663-, 2943-, 1671-, 657-, 641-, 796-, and 814-cm^{-1} Modes of CHD, AAP, HOAP, DEAM, ISOV, DODO, MA, PTSO, and VAN, Respectively

Material	Mode (cm^{-1})	Γ (cm^{-1})	N_R (Photons/s.cm.sr.mW)	σ_R (cm^2)
CHD	1174	8.4 ± 0.2	$4.90 \pm 0.25 \times 10^{7}$	$1.47 \pm 0.12 \times 10^{-28}$
AAP	714	4.4 ± 0.1	$8.63 \pm 0.42 \times 10^{6}$	$1.67 \pm 0.13 \times 10^{-29}$
HOAP	1663	4.5 ± 0.1	$2.44 \pm 0.12 \times 10^{7}$	$5.15 \pm 0.40 \times 10^{-29}$
DEAM	2943	10.6 ± 0.2	$1.43 \pm 0.07 \times 10^{7}$	$3.25 \pm 0.26 \times 10^{-29}$
ISOV	1671	4.5 ± 0.1	$1.04 \pm 0.05 \times 10^{8}$	$1.57 \pm 0.13 \times 10^{-28}$
DODO	657	2.7 ± 0.1	$1.50 \pm 0.08 \times 10^{7}$	$2.07 \pm 0.17 \times 10^{-29}$
MA	641	6.8 ± 0.2	$5.17 \pm 0.26 \times 10^{6}$	$1.00 \pm 0.08 \times 10^{-29}$
PTSO	796	5.7 ± 0.2	$8.15 \pm 0.41 \times 10^{6}$	$3.73 \pm 0.30 \times 10^{-29}$
VAN	814	5.2 ± 0.1	$6.16 \pm 0.31 \times 10^{6}$	$1.42 \pm 0.11 \times 10^{-29}$

5. SUMMARY

We have measured the values of the absolute Raman cross sections of several chemicals for 785-nm excitation with an accuracy of ±9% for the liquids and ±13% for the powders. The measured values of the Raman cross sections are of the order of 10^{-28} to 10^{-29} cm^2.

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14. ABSTRACT

We have measured the Raman spectra of several chemicals for 785-nm excitation using a Raman spectrometer with focal length of 500 mm, f-number of 6.5, and resolution of 2.0 ± 0.1 cm^{-1}. Values of $1.92 \pm 0.12 \times 10^{-29}$, $2.50 \pm 0.15 \times 10^{-29}$, $3.10 \pm 0.19 \times 10^{-28}$, $1.17 \pm 0.07 \times 10^{-29}$, and $1.53 \pm 0.09 \times 10^{-29}$ cm^2 have been determined for the Raman cross section σ_R of neat Diethyl phthalate (DEP), dimethyl phthalate (DMP), ethyl cinnamate (EC), propylene carbonate (PC), and tripropylene phosphate (TPP) for the Raman modes at 1041, 1040, 1638, 849, and 726 cm^{-1}, respectively. The values of the linewidth (FWHM) Γ for these modes have been determined to be 6.6 ± 0.1, 7.0 ± 0.1, 11.3 ± 0.2, 15.8 ± 0.2, and 29.0 ± 0.3 cm^{-1}, respectively. Values of $1.47 \pm 0.12 \times 10^{-28}$, $1.67 \pm 0.13 \times 10^{-29}$, $5.15 \pm 0.40 \times 10^{-29}$, $3.25 \pm 0.26 \times 10^{-29}$, $1.57 \pm 0.13 \times 10^{-28}$, $2.07 \pm 0.17 \times 10^{-29}$, $1.00 \pm 0.08 \times 10^{-29}$, $3.73 \pm 0.30 \times 10^{-29}$, and $1.42 \pm 0.11 \times 10^{-29}$ cm^2 have been determined for σ_R for the powders of 1,3-cyclohexanedione (CHD), 3'-aminoacetophenone (AAP), 3'-hydroxyacetophenone (HOAP), diethyl acetamidomalonate (DEAM), isovanillin (ISOV), lactide (DODO), Meldrum's acid (MA), p-tolyl sulfoxide (PTSO), and vanillin (VAN) for the Raman modes at 1174, 714, 1663, 2943, 1671, 657, 641, 796, and 814 cm^{-1}, respectively. The values of Γ for these modes have been determined to be 8.4 ± 0.2, 4.4 ± 0.1, 4.5 ± 0.1, 10.6 ± 0.2, 4.5 ± 0.1, 2.7 ± 0.1, 6.8 ± 0.2, 5.7 ± 0.2, and 5.2 ± 0.1 cm^{-1}, respectively.

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